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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: METHOD OF PROVIDING AN ESSENTIAL OIL EXTRACT OF CAPSICUM, AND THE EXTRACT

(57) Abstract: A method of providing an essential oil extract of capsicum, and the extract itself, are provided. The extract contains capsaicinoid and terpene. Capsicum from which seeds and stems have preferably been removed is mixed, in powder form, with a solvent, especially pentane, to dissolve at least some of the capsicum. During or after the mixing step, the liquid solution of capsicum in the solvent is brought to a temperature of less than or equal to 64°C to significantly reduce the solvent content thereof and produce an essential oil extract that contains capsaicinoid and terpene, especially in the naturally occurring ratio thereof.

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METHOD OF PROVIDING AN ESSENTIAL OIL EXTRACT OF
CAPSICUM, AND THE EXTRACT

Technical Field

5 The present invention relates to a method of providing an effective essential oil extract of capsicum that contains the natural capsaicinoid to terpene ratio for use in culinary, medicinal, and repellent formulations. The present invention also relates to the extract itself.

10 Terpenes are unsaturated hydrocarbon molecules composed of two or more isoprene units. The two most abundant natural sources of terpenes are turpentine and other essential oils. [TERPENOIDS, pp.709-710] Terpenes recovered from botanical sources have historically been used in adhesives, inks, coatings, and rubber. [AIGER]

15 Essential oils have been isolated from botanical sources for use in flavors, fragrances, and medicine since antiquity. The products derived from essential oils have large volume usage for specific applications. According to the Encyclopedia of Chemical Engineering, "Essential oils are concentrated, rectified, extracted, or chemically treated to further isolate vital components, purify, adjust
20 properties, or increase the concentration of significant flavor or fragrance components." [p. 309] The largest class of components that constitute the essential oil is the terpene. Whole or partial removal of nonflavor or undesirable components such as the terpenes yields a
25 concentrated or folded oil. [Encyclopedia of Chemical Engineering, ESSENTIAL OIL, p. 309] "Although termed concentration, this process is, nevertheless, not merely a concentration in the ordinary sense, since the flavor body of the concentrate is always weaker than that of the complete essential oil, demonstrating that valuable products are

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lost in the course of removing the terpenes." [Encyclopedia of Chemical Engineering, ESSENTIAL OIL, p. 310]

Processing methods for isolation and extraction of desired components from the essential oil may include solid-liquid or solid-vapor extraction, liquid-liquid or liquid-vapor extraction, and supercritical liquid extraction. In some cases both distillation and solvent extraction are needed for complete removal of terpenes. The basis for success of extraction processes is the difference in affinity for one component or material over another. [Encyclopedia of Food Science and Technology, EXTRACTION, p. 791-793]

Capsaicin (8-methyl-N-vanillyl-nonenamide), isolated by Thresh in 1878 [THRESH], is the parent molecule of similarly structured alkaloids responsible for the pungent active principal in a variety of capsicum species. Historically, the cultivation of capsicum, and subsequent manufacture of capsicum products, for commercial use in culinary, medicinal, and repellent formulations has been primarily based on capsaicin content. [BOSLAND] Oleoresin capsicum is perhaps the most widely used product in the aforementioned formulations. Oleoresin capsicum is the alcoholic fraction of the ether extract of capsicum. In this process, alcohol is added to the ether (non-polar) extract and further distilled to separate and isolate capsaicinoids from undesired components.

Tandon [JOURNAL OF FOOD SCIENCE, p. 5] states, "In the industry, whole chili powder (intact with stems and seeds) is used for the manufacture of oleoresin of capsicum. The fatty-oil, which is recovered as a by-product and is rich in color (terpenes), is a waste product at present. Further, separation of this oil from the extract to recover the oleoresin, is an elaborate process." The art teaches the removal of seeds and stems from whole capsicum used in the

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manufacture of oleoresin capsicum. This method eliminates the undesired fatty-oil components associated with the seeds and stems, facilitating the removal of color matter (terpenes) by a simple percolation method of extraction to produce a purer oleoresin.

5 [TANDON] In the percolation method of extraction, a properly ground botanical is placed in an extractor with a removable bottom and a filter bed. The solvent is percolated either with or without heat for a predetermined period of time. The extract is drained and the solvent recovered by distillation and recycled. [Encyclopedia of Chemical

10 Engineering, Vol. 16, pp. 314] The art lists ether, hexane, chloroform, alcohol, and acetone as solvents preferred for the production of oleoresin capsicum.

The art does not teach the retention of the naturally occurring capsaicinoid to terpene ratio.

15 Veech [US Patent 5,629,045] discloses methods of extracting capsaicin from cayenne pepper for use as a repellent in wood and marine coating applications. Veech states, "Whole peppers from the capsicum genus...are extracted for their constituent capsaicinoids...." [Embodiment 2, p. 8 of 10]

20 The art teaches that the capsaicinoids can be solvent extracted and subsequently partitioned from the dry plant with organic solvents including petroleum ether, alcohol, ethyl acetate, acetone, chloroform, methyl chloride, linseed oil, and supercritical fluids such as carbon dioxide. The process disclosed in the art involves two separate

25 operations. First, capsicum powder and an organic solvent (polar or nonpolar) are heated under reflux using the Soxhlet extraction method (i.e., a modified, scaled-down version of the percolation method of extraction). Second, a solvent of opposite polarity (polar or nonpolar) is added to the extracted solution to further partition undesired

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components (nonpolar) and concentrate the desired capsaicinoid components. The process yields an oleoresin capsicum that may be mixed with wood or marine coatings. The art does not teach the retention of the naturally occurring capsaicinoid to terpene ratio.

5 Etscorn et al [US Patent 5,674,496] discloses a process that produces an essential oil solution for use in repellent applications. The patent lists several commercially available volatile organic solvents for use in the simple extraction process. The compatibility of the solvent with each coating material determines what solvent is selected
10 for the extraction process. The preferred solvent serves in the extraction process, and facilitates the loading of each coating material with the repellent extract.

 The art further discloses methods of increasing the potency of the repellent extract. First, commercially available habanero
15 ground powder (constituents include seeds and stems) and a commercial volatile organic solvent are mixed/blended while heated. To promote potency, the mixture sits undisturbed between episodes of mixing, blending, and heating. The mixture is filtered, and the filtered solution is then combined with additional habanero powder and
20 solvent. The extraction process is again repeated, resulting in a "double-treated" extract solution. The art teaches heating the mixture at a preferred temperature range of 60°C to 75°C prior to filtration.

 The resultant extract/solvent solution is added to various coating materials. The examples provided in the art demonstrate that
25 the extract/solvent solution constitutes 25% to 35% of the total repellent coating material.

 The art does not teach the removal of non-essential components from the extract, which components are attributable to the grounded seeds and stems in the habanero powder used. The art

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does not teach the use of a universal solvent in the extraction process. Further, the art does not teach heating the repellent solution after filtration to eliminate solvent content. Yet further, the art does not disclose a temperature range of less than or equal to 64°C for the retention of the natural capsaicinoid to terpene ratio characteristic of the essential oil.

The extract solution disclosed by Etscorn et al contains impurities or non-essential components that will have a negative impact on the efficacy and integrity of the repellent coating material. Additionally, the use of a different solvent for each specific extraction could be expensive and impractical in the industrial manufacture of the extract. Further, a repellent coating material containing 25% to 35% volatile organic solvent may violate federal laws limiting the use of volatile organic compounds in architectural coatings [Federal Register - EPA]. Finally, the preferred temperature range disclosed in the art of up to 75°C will adversely impact the extract/solvent solution.

It is an object of the present invention to provide an effective means for extracting a substantially pure essential oil from capsicum, especially in an economical and efficient manner. Such an extract is intended in particular for medicinal, culinary, and repellent formulations. A further object of the present invention is to provide an effective means for significantly reducing the amount of volatile organic solvent required, if at all, to incorporate the extract into various formulations.

Disclosure of the Invention

The method of the present invention is characterized primarily by: providing, in powder form, capsicum from which seeds and stems have been removed; mixing the capsicum with a solvent to dissolve at least some of the capsicum; if desired, separating a liquid

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solution of capsicum in the solvent from any solid or non-dissolved material; and during or after the mixing step bringing the liquid solution to a temperature of 64°C or less by applying heat if necessary, to significantly reduce the solvent content thereof and produce an essential oil extract that contains capsaicinoid and terpene.

With regard to the heating step, as indicated heat can be applied to the liquid solution during or after the step of mixing the capsicum with the solvent. In particular, such heating could be accomplished in a closed system, for example by the percolation or Soxhlet methods of extraction, with the latter using a water bath. Distillation could also be used, in which case the heat source is preferably of a non-flame type. Where the solvent is under pressure, it may be sufficient to operate at atmospheric pressure to achieve evaporation of the solvent.

It is also to be understood that the time required for heating will of course vary as a function of the solvent that is used. In addition, a partial vacuum or other suitable aid could be utilized to reduce the time needed for removing the solvent or reducing the residual content thereof.

The invention relates to a capsicum extract that exploits the effects of capsaicinoids and capitalizes on the inherent adhesive properties of the terpenes. The extract preferably retains the naturally occurring capsaicinoid to terpene ratio of the starting capsicum material.

The invention provides modes for producing a pure essential oil for use in repellent, medicinal, and culinary formulations. The extract of the invention is prepared by grinding capsicum that has been deseeded/destemmed, for example by coring the capsicum. Examples of sources of capsicum include, as non-limiting examples,

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habanero pepper, cayenne pepper and African birdseye pepper. The appropriate pepper will of course depend upon the application for which the extract is intended. Any fungus that is present should also be removed, for example by scraping the capsicum pod. The capsicum powder is mixed/blended in any suitable manner with an organic solvent, and may then be filtered. The filtered solution is heated for example from 1 to 16 hours, to a temperature of less than or equal to 64°C. This temperature facilitates total evaporation of the solvent from the filtered solution without destroying the terpenes. Non-polar solvents with proximate boiling points heated to less than or equal to 64°C, such as hexane (b.p. about 69°C), or preferably pentane (b.p. 36°C), may be used in the extraction process, although if hexane is used a far longer heating time is required at the prescribed temperature of no greater than 64°C since this temperature is less than the boiling point of hexane. Other solvents, such as butane and propane, could also be used, as well as any other non-polar solvent having a boiling point of $\leq 64^{\circ}\text{C}$. With pressurized solvents, the heating step could even be eliminated.

The combination of temperature range, low boiling-point non-polar solvents, and deseeded/destemmed capsicum powder provides a potent and effective pure essential oil extract. The resultant extract significantly reduces the introduction of volatile organic solvents when the extract is used in various formulations.

Because non-essential components are eliminated by destemming/deseeding the capsicum and all essential oil components are retained during the extraction process, the extract is compatible with various polymeric formulations.

An advantage of the invention is the retention of the natural capsaicinoid to terpene ratio found in the essential oil.

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A further advantage is the removal of non-essential components contributed by the seeds and stems by mechanical, chemical, or genetic methods.

5 A further advantage is the efficacy and compatibility of the extract with a wide range of formulations.

It should be noted that none of the prior art teaches or suggests the retention of the naturally occurring capsaicinoid to terpene ratio.

Detailed Description of Preferred Embodiments

10 The invention is based on the retention of the natural capsaicinoid to terpene ratio that constitutes the essential oil of capsicum. The present invention exploits the effect of capsaicinoids and capitalizes on the inherent adhesive properties of the terpenes. The removal of non-essential components attributable to the seeds and
15 stems, and more importantly the retention of essential components during the extraction process, renders an inexpensive and effective extract compatible to a wide range of polymeric products. The extract may be used in various repellent, medicinal, and culinary formulations.

In the preferred embodiment of the invention,
20 deseeded/destemmed capsicum powder and pentane (b.p. 36°C) are blended/mixed and then filtered. The filtered solution is heated to 36°C to evaporate the solvent content. In a closed system the solvent may be recovered and reused. A heating range of 65°C or more adversely impacts the natural capsaicinoid to terpene ratio. The volatilization of
25 essential components, especially partial or whole elimination of terpenes, will have a negative impact on the efficacy, integrity and compatibility of the extract. Therefore, the inventive temperature of less than or equal to 64°C, and especially less than 60°C, is critical to retaining the naturally occurring capsaicinoid to terpene ratio. Such a

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temperature is sufficient for removing solvent to a residual content thereof of less than or equal to 1% by volume.

The extract rendered by the present invention may be incorporated into various formulations with minimal use of volatile organic solvents.

Example 1

Twenty pounds of whole dry habanero pepper (Pendry's, 1221 Manufacturing St., Dallas, Texas 75207) was divided into two, ten pound samples.

The seeds and stems were removed by hand from one of the samples, sample A, and then discarded. The pericarp (approximately 4.5 lbs. in weight) was collected and ground into powder for extraction. The other sample, with stems and seeds intact, was ground into powder and then separated into two, five pound samples B and C for extraction.

The following extraction procedure was used to process each sample separately.

One-half pound of ground habanero and five hundred milliliters of pentane (Industrial Chemical, 11722 Charles St., Houston, Texas 77041) were placed in an industrial blender and blended for two minutes. The blended contents were poured into a common container and sealed. The process was repeated until the sample was exhausted. The container was sealed and allowed to set for eight hours. (This allowed the solid particles to separate from the solution for easier filtration.) The solution was filtered into a second, clean container and then placed unsealed in a water bath (less than 60°C for sample A and B, or $\geq 70^{\circ}\text{C}$ for sample C) to reduce solvent content.

Samples A and B were placed in the water bath for sixteen hours and yielded one hundred milliliters of habanero extract.

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Sample C was placed in the water bath for two hours and yielded seventy-five milliliters of habanero extract.

5 Tests on the extracts showed that Sample A had a higher percentage of nonvolatile oil than did the other two samples. In addition, the Scoville Heat Units of Sample A were significantly greater than for Sample B and Sample C.

10 It should also be noted that the non-essential stem/seed components found in Samples B and C will adversely affect the chemical compatibility of the extract with polymeric materials.

The results also demonstrate that removal of stem/seed components in Sample A, combined with a low boiling point extraction, will yield a pungent extract without elimination of the terpene content.

15 The present invention is, of course, in no way restricted to the specific disclosure of the specification, but also encompasses any modifications within the scope of the appended claims.

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CLAIMS:

1. A method of providing an essential oil extract of capsicum, wherein the extract contains capsaicinoid and terpene, said method characterized by the steps of:

5 providing, in powder form, capsicum from which seeds and stems have been removed;

mixing said capsicum with a solvent to dissolve at least some of the capsicum; and

10 during or after said mixing step, bringing a liquid solution of capsicum in said solvent to a temperature of $\leq 64^{\circ}\text{C}$ to significantly reduce the solvent content thereof and produce an essential oil extract that contains capsaicinoid and terpene.

2. A method of providing an essential oil extract of capsicum, wherein the extract contains capsaicinoid and terpene, said method characterized by the steps of:

15 providing, in powder form, capsicum from which seeds and stems have been removed;

mixing said capsicum with pentane to dissolve at least some of the capsicum; and

20 during or after said mixing step, bringing a liquid solution of capsicum in said pentane to a temperature of $\leq 64^{\circ}\text{C}$ to significantly reduce the pentane content thereof and produce an essential oil extract that contains capsaicinoid and terpene, wherein said essential oil extract contains the naturally occurring capsaicinoid to terpene ratio.

25 3. A method of providing an essential oil extract of capsicum, wherein the extract contains capsaicinoid and terpene, said method characterized by the steps of:

providing, in powder form, capsicum from which

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seeds and stems have been removed;

mixing said capsicum with a solvent to dissolve at least some of the capsicum; and

5 during or after said mixing step, bringing a liquid solution of capsicum in said solvent to a temperature of $\leq 64^{\circ}\text{C}$ to significantly reduce the solvent content thereof and produce an essential oil extract that contains capsaicinoid and terpene, wherein said essential oil extract contains the naturally occurring capsaicinoid to terpene ratio, and wherein said liquid solution is heated until the
10 solvent content thereof is reduced to $\leq 1\%$ by volume.

4. A method of providing an essential oil extract of capsicum, wherein the extract contains capsaicinoid and terpene, said method including the steps of:

15 providing capsicum in powder form;
mixing said capsicum with a solvent to dissolve at least some of the capsicum; and

during or after said mixing step, bringing a liquid solution of capsicum in said solvent to a temperature of $\leq 64^{\circ}\text{C}$ to significantly reduce the solvent content thereof and produce an
20 essential oil extract that contains capsaicinoid and terpene.

5. A method according to any of claims 1-4, characterized in that said essential oil extract contains the naturally occurring capsaicinoid to terpene ratio.

25 6. A method according to claim 5, characterized in that said solvent is selected from the group consisting of hexane, pentane, butane, propane and other non-polar solvents having a boiling point of $\leq 64^{\circ}\text{C}$, especially pentane.

7. A method according to any of claims 1-6, characterized in that said step of providing capsicum in powder form

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comprises removing stems and seeds from capsicum pods and then grinding said pods, and can include the further step of removing fungus from said pods prior to grinding same.

5 8. A method according to any of claims 1-7, characterized in that said liquid solution is heated until the solvent content thereof is reduced to $\leq 1\%$ by volume, wherein said liquid solution is heated for 1 to 16 hours, for example in a hot water bath to a temperate of less than 60°C .

10 9. A method according to any of claims 1-8, characterized in that said liquid solution is brought to atmospheric pressure, and/or by the further step of separating a liquid solution of capsicum in said solvent from any solid or non-dissolved material, for example by filtering to remove any solid or non-dissolved material.

15 10. An essential oil extract of capsicum produced according to the method of any of claims 1-9, characterized in that said extract contains the naturally occurring capsaicinoid to terpene ratio, and/or in that said extract has a residual solvent content of $\leq 1\%$ by volume.

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : A61K 35/78

US CL : 424/760

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 424/760

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WEST

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,674,496 A (ETSCORN et al) 07 October 1997, col 3, line 22 - col 9, line 60.	1-6
Y	US 6,066,060 A (BELGOROD) 09 May 2000, col 2, line 1 - col 5, line 5.	1-6
Y	Database EPAB on WEST, No. FR2721213A1. NEGOL et al. 'Capsicum frutescens extract prepn.' 22 December 1995, Abstract.	1-6

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

*	Special categories of cited documents	"T"	later document published after the international filing date or priority date and in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"B"	earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L"	document which may throw doubt on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"a"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

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Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This international report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☐ Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
3. ☒ Claims Nos.: 7-10
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.